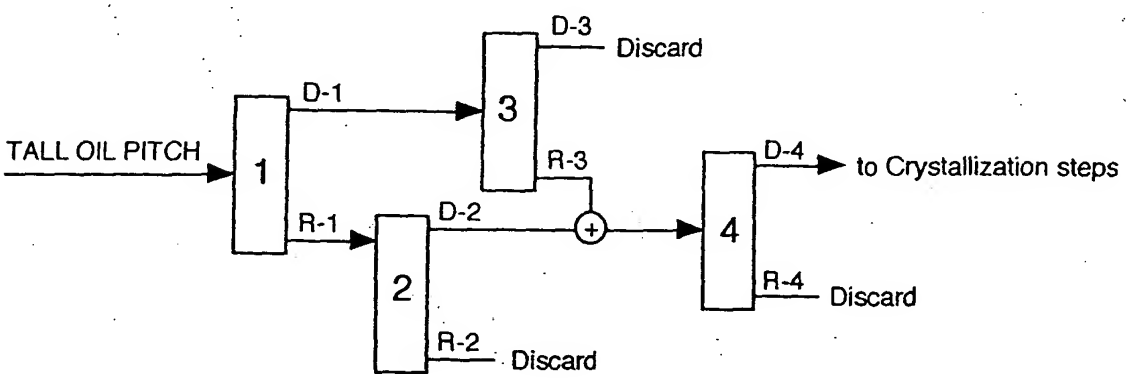
It is respectfully submitted that the inclusion of the word "only" in steps (c) and (d) of amended claim 1 clearly and definitively excludes the distillation and dissolution process as suggested in D1. For ease and reference, the distillation steps of Malik et al. are depicted below:



Malik et al. Distillation Steps

Note: A similar drawing was included with the applicants letter of 24/09/1999. In the earlier drawing, distillation stages 2 and 3 were inadvertently switched such that the drawing did not correspond with references to (D–2), (R–2), (D–3) and (R–3) added to the applicant's translated version of CS 256 092. This has been corrected in the above drawings.

Step (c) of amended claim 1 speaks of "distilling only said bottom fraction in a second evaporator to produce a light phase distillate". If R–1 of Malik et al. is regarded as the bottom fraction, and if D–2 is regarded as the light phase distillate, then Malik et al. fail to satisfy claim 1 because step (d) of claim 1 requires "dissolving only said light phase distillate is a solvent. Malik et al. do not dissolve "only said" light phase distillate in a solvent. The substance they dissolve is a subsequent distillate D-4 produced by evaporator 4. Moreover, the input to evaporator 4 is not "only said" light phase distillate. It is the product produced by the combination of D–2 and R–3, the latter being a bottom fraction from a parallel distillation path.

As defined in claim 1, the steps of distillation and dissolution are a sequence of steps constrained to follow in series without any recombinant parallel paths of product flow as in the case of Malik et al. It is respectfully submitted that the language of claim 1 admits to no other interpretation.

Similar observations may be made with respect to amended claim 22. Claim 22 is based upon the teachings of Examples 4 and 5 (page 10, line 15 et seq.) where a sample of light phase distillate was re-distilled to achieve an increase in crystal purity. Upon comparison between the purities set forth in Tables 3 and 5, it will be seen that the increase is marginal over an already high purity. However, claim 22 is nevertheless considered

important because the presence of the re-distillation step called for by claim 22 might be considered to take the process outside the ambit of amended claim 1.

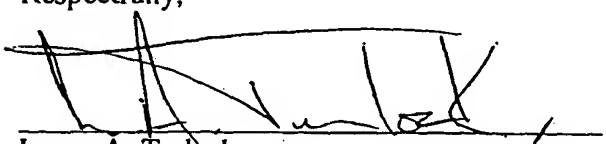
Despite the addition of the re-distillation step in amended claim 22, the overall steps of distillation and dissolution in claim 22 remain as a sequence of steps constrained to follow in series as in the case of amended claim 1. The use of the word "only" in the language of the claim positively excludes any recombinant parallel paths as appear in the case of Malik et al.

Together with the foregoing clarification, the Examiner is respectfully referred back to remarks contained in the applicant's letter of 24/09/1999, particularly the "Key observations" concerning Malik et al. (reference D1) made on page 4 of that communication. In short, the present invention is considered to offer significant advantages over the process disclosed in D1.

In summary, it is respectfully submitted that the claims as amended clearly are not anticipated by D1 and that all objections based upon lack of novelty should be withdrawn in view of the foregoing amendments and arguments. With regard to the opinion expressed in the case of claims 8, 9, 17, 18, 23, 24 (lack of inventive step), it is respectfully submitted that the objections raised should likewise be withdrawn because all such objections depend upon the efficacy of the objections discussed above.

Favorable consideration is respectfully solicited in view of the amendments and arguments that have been made. Pursuant to Rule 66.4(b), the applicants respectfully request that they be given one or more additional opportunities to submit further amendments and/or arguments, should the amendments and/or arguments submitted herewith be deemed insufficient.

Respectfully,



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REPLACEMENT PAGES SUBMITTED WITH RESPONSE TO FIRST WRITTEN OPINION

- Pages 3, 3a and 4 of description
- Pages 14, 16 and 17 of claims

In U.S. Patent No. 5,097,012 granted on 17 March 1992, Thies et al. disclose a method for the isolation of sterols from crude tall oil by water extraction at elevated temperatures and pressures.

In U.S. Patent No. 3,943,117 granted on 9 March 1976, Force discloses a process for saponifying tall oil pitch in which a water-soluble cationic amine is used in conjunction with an alkali. In U.S. Patent No. 4,524,024 granted on 18 June 1985, Hughes teaches the hydrolysis of tall oil pitch at elevated temperatures to increase the recovery of fatty acids from tall oil pitch. In U.S. Patent No. 3,887,537 granted on 3 June 1975, Harada et al. disclose the recovery of fatty acids and rosin acids from tall oil pitch by first saponifying tall oil pitch with an alkali metal base and a low molecular weight alcohol, and then introducing the reacted mixture into a thin film evaporator to remove low-boiling matter such as water, alcohol use and light unsaponifiables. The bottom fraction from the first evaporator is next fed to a second thin film evaporator in which the unsaponifiables including sterols are removed as the light ends and a molten soap is recovered as the bottom fraction. Fatty acids and rosin acids are recovered from the molten soap fraction by acidulation conventionally with a mineral acid. In U.S. Patent No. 3,926,936 granted on 16 December 1975, Lehtinen teaches the recovery of fatty acids and rosin acids from tall oil pitch by reacting tall oil pitch with an alkali at 200 to 300 degrees Celsius, in the amount of 5 to 25% of tall oil pitch, prior to vacuum distillation of the heated mixture to recover the fatty acids and rosin acids in the distillate fraction.

Reference is also made to Chemical Abstracts, vol. 112, no. 20, 14 May 1990, Columbus, Ohio US; abstract no. 181758, MALIK, Lubomir et al: "Isolation of phytosterols from tall - oil rosin", XP002104877 & CS 256 092 A (Czech). Malik et al. disclose a process for extracting phytosterols which includes the use of four distillation stages. Product flow is split into parallel distillation paths in a first distillation stage then, following further distillation in each parallel path, is partially recombined prior to a final distillation stage. To achieve high purity phytosterols, the output from the final distillation

stage is subjected to two stages of crystallization utilizing relatively large amounts of solvent.

SUMMARY OF THE INVENTION

In a broad aspect of the present invention there is provided a new and improved method of preparing phytosterols from tall oil pitch containing steryl esters, the method comprising the steps of:

(a) converting the steryl esters to free phytosterols while in the pitch to produce a modified pitch containing the free phytosterols;

(b) distilling the modified pitch in a first evaporator to remove light ends from the modified pitch and produce a bottom fraction containing the free phytosterols;

(c) distilling only the bottom fraction in a second evaporator to produce a light phase distillate containing the free phytosterols;

(d) dissolving only the light phase distillate in a solvent comprising an alcohol to produce a solution containing the free phytosterols;

(e) cooling the solution to produce a slurry with the free phytosterols crystallized in the slurry; and,

(f) washing and filtering the slurry to isolate the crystallized phytosterols.

Preferably, the step of converting the steryl esters to free phytosterols comprises the steps of saponifying the tall oil pitch with an alkali metal base, neutralizing the saponified pitch with an acid, and heating the neutralized pitch to remove water. The resulting pitch with such water removed defines the modified pitch.

Unlike the process of Malik et al., the foregoing process enables the preparation of high purity phytosterol crystals from tall oil pitch with only two distillation stages and only one stage of crystallization, and to do so with the use of a comparatively small amount of solvent. Nevertheless, it may be considered desirable in some cases to achieve phytosterol yields with even higher crystal purity. In accordance with another embodiment of the invention, a marginal improvement is achieved as follows:

(a) producing a light phase distillate containing free phytosterols in the manner described in steps (a) to (c) above;

(b) re-distilling only the light phase distillate so produced to enhance the concentration of free phytosterols in the light phase distillate;

(c) dissolving only the re-distilled light phase distillate in a solvent comprising an alcohol to produce a solution containing the free phytosterols; and,

(d) continuing the procedure as in steps (d) and (f) above to isolate crystallized phytosterols.

Although this procedure involves additional distillation steps, the amount of alcohol required during the crystallization stage remains small compared to the case of Malik et al.

BRIEF DESCRIPTION OF THE DRAWINGS

The FIGURE shows a schematic flow diagram for the preparation of high purity phytosterol crystals from tall oil pitch in accordance with the present invention.

DESCRIPTION OF PREFERRED EMBODIMENT

In accordance with the present invention, the isolation of phytosterols from tall oil pitch first requires converting steryl esters present in the pitch to free phytosterols while in the pitch. The result is a modified pitch containing free phytosterols.

It is contemplated that the required conversion may be accomplished by various methods. In the FIGURE, the conversion step is indicated by block 30 (shown in broken outline) which receives an incoming feed of tall oil pitch 1 and produces modified pitch 11 as an output. The presently preferred method of conversion involves the use of an alkali base treatment and is indicated by the elements contained within block 30.

WE CLAIM:

1. A method of preparing phytosterols from tall oil pitch (1) containing steryl esters, said method comprising the steps of:

(a) converting said steryl esters to free phytosterols while in said pitch to produce a modified pitch (11) containing said free phytosterols;

(b) distilling said modified pitch (11) in a first evaporator (12) to remove light ends (13) from said modified pitch and produce a bottom fraction (14) containing said free phytosterols;

(c) distilling only said bottom fraction (14) in a second evaporator (15) to produce a light phase distillate (16) containing said free phytosterols;

(d) dissolving only said light phase distillate (16) in a solvent (21) comprising an alcohol to produce a solution containing said free phytosterols;

(e) cooling said solution to produce a slurry (19) with said free phytosterols crystallized in said slurry; and,

(f) washing and filtering said slurry (19) to isolate said crystallized phytosterols (22).

2. A method as defined in claim 1, wherein said modified pitch (11) comprises less than 1% water by weight.

3. A method as defined in claim 1 or 2, wherein said solvent (21) comprises a low molecular weight monohydric alcohol.

4. A method as defined in claim 1 or 2, wherein said solvent (21) comprises a low molecular weight monohydric alcohol and water.

5. A method as defined in claim 1 or 2, wherein said slurry (19) is washed and filtered using a solvent like said solvent (21) used to dissolve said light phase distillate.

6. A method as defined in claim 1, wherein said step of converting said steryl esters to free phytosterols comprises the steps of:

(a) saponifying said tall oil pitch (1) with an alkali metal base (2);

14. A method as defined in claim 6, wherein said neutralized pitch has a water phase pH in the range of 4 to 7.

15. A method as defined in claim 6, wherein said heating step comprises heating at a temperature in the range 90 to 100 deg. C for a time sufficient to effect the bulk disengagement of water from the organic phase.

16. A method as defined in claim ~~15~~, wherein said heating step further comprises heating under vacuum conditions such that said modified pitch (11) comprises less than 1% water by weight.

17. A method as defined in claim 1 or 6, wherein said light ends are removed in a wiped film evaporator (12) operating at a pressure in the range of 0.1 to 10 millibars and at a temperature in the range 160 to 280 deg. C.

18. A method as defined in claim 1 or 6, wherein said bottom fraction is evaporated in a wiped film evaporator (15) operating at a pressure in the range of 0.01 to 1.0 millibars and at a temperature in the range 180 to 300 deg. C.

19. A method as defined in claim 6, wherein said solvent (21) comprises a low molecular weight monohydric alcohol.

20. A method as defined in claim 6, wherein said solvent (21) comprises a low molecular weight monohydric alcohol and water.

21. A method as defined in claim 1 or 6 in which the crystallization of phytosterols is effected at a temperature in the range of 0 to 35 deg. C.

22. A method of preparing phytosterols from tall oil pitch (1) containing steryl esters, said method comprising the steps of:

(a) converting said steryl esters to free phytosterols while in said pitch to produce a modified pitch (11) containing said free phytosterols;

(b) distilling said modified pitch (11) in a first evaporator (12) to remove light ends (13) from said modified pitch and produce a bottom fraction (14) containing said free phytosterols;

(c) distilling only said bottom fraction (14) in a second evaporator (15) to produce a light phase distillate (16) containing said free phytosterols;

(d) re-distilling only said light phase distillate (16) to enhance the concentration of free phytosterols in said light phase distillate;

(e) dissolving only said re-distilled light phase distillate in a solvent (21) comprising an alcohol to produce a solution containing said free phytosterols;

(f) cooling said solution to produce a slurry (19) with said free phytosterols crystallized in said slurry; and,

(g) washing and filtering said slurry (19) to isolate said crystallized phytosterols (22).

23. A method as defined in claim 22, wherein said solvent (21) further comprises water added in a proportion up to 35% by weight relative to the organic solvent phase.

24. A method as defined in claim 23, wherein the weight ratio of solvent to distillate is between 0.3 to 2.0.

25. A process according to claim 19, 20 or 24 in which the alcohol is selected from:

(a) methanol;

(b) ethanol;

(c) 2-propanol;

(d) a combination of alcohols comprising two or more of methanol, ethanol and 2-propanol.

26. Phytosterols prepared from tall oil pitch in accordance with the method as defined in any one or more of the preceding claims.